SYNTHESIS AND CHARACTERIZATION OF CERAMIC (ALUMINA) MEMBRANE SHEET

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INTRODUCTION

Separation processes occupy a key position in the chemical process industries, only next to chemical reactions, and have developed for centuries in the form of conventional separation techniques such as distillation, solvent extraction, crystallization or filtration . These were enriched in the 1960's by another class of processes based on permselective membranes. These are in many cases, faster, more efficient and more economical than conventional processes. Most of the commercially available membranes are based on organic polymers which are generally stable only at temperatures below 100 C, narrow pH range and in the absence of organic solvents. Ceramic (alumina) membranes , also called 3rd generation membranes exhibit advantageous characteristics- physical , chemical and thermal stability and longer lifetime [1-4] .

Macroporous (pore diameter larger than 50 nm) symmetric ceramic membranes can be prepared by cold isostatic pressing of dry alumina powder with addition of binders. After burning away the organic material, the compact is sintered and the porosity can be adjusted by the sintering temperature. The membranes thus produced ,can be used for microfiltration in liquid media separations and can serve as an ideal defect-free support for the production of UF, hyperfiltration or/and gas separation membranes.

The objectives of the present study were to synthesize ceramic alumina membranes by the moulding (pressing) process and determine the effect of pressing force and the sintering temperature on the characteristics of the membrane : density , porosity and pore size distribution.

EXPERIMENTAL

The starting material for the membrane were powdered aluminum oxide(Al_2O_3) and alumina granules (99.7% Al_2O_3; crystal size, 1 μm ;

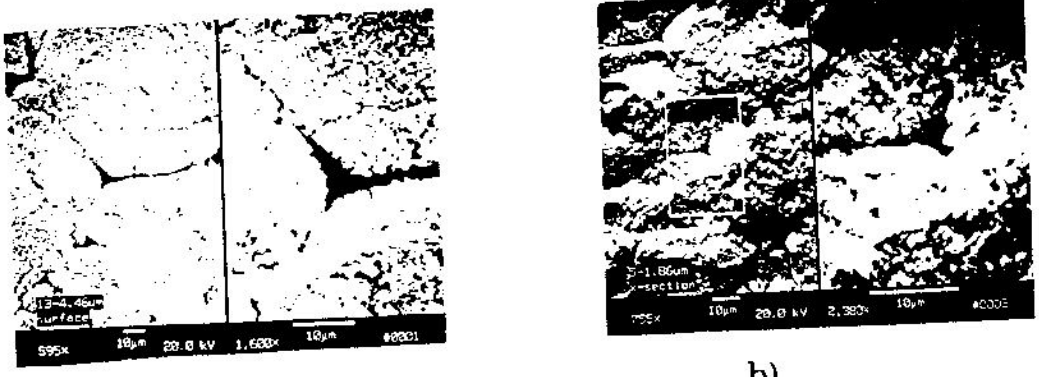
granule size 60-80 μm) . Alumina granules were prepared by spray drying of alumina with additives and binders (ethylene glycol and PVA). Particle size analysis were done by HORIBO size distribution analyzer. The fabrication of the membrane sheet was done in a hardened steel rectangular die (dimension 57.0 X 34.2 mm) using DORST automated powder press with pressing force in the range 115-145 kN .The minimum thickness of the sheet produced was 0.5mm . Circular membranes were produced using Manual Hydraulic Press. The membranes were characterized for the microstructure and pore size distribution using Scanning Electron Microscope (SEM) , and density and porosity using water absorption measurement [5].

RESULTS AND DISCUSSION

Size, shape and surface roughness has important bearing on the characteristics of membrane produced. Free flowing granular form of alumina was found to be a necessary requirement for uniform membrane sheet. All the membrane produced possessed rigid body and crack-free smooth surface.

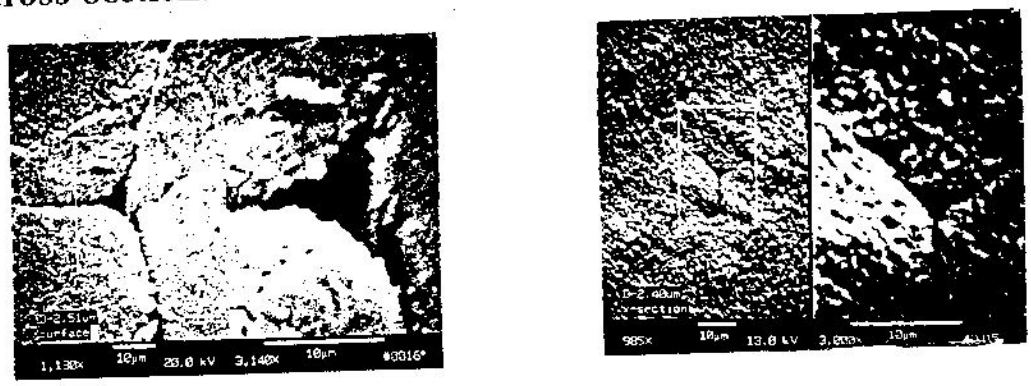
The microstructure of the membrane sheets at various conditions of pressing force and sintering temperature were analyzed from SEM micrographs. Figures 1 and 2 show typical micrographs and indicate that the bodies are porous; the pore size of the membrane shrink as the pressure and sintering temperature are increased, however the strength and particle-particle bonding also increases.

The effect of moulding pressure and sintering temperature on membrane porosity and density are shown in figure 3a and 3b respectively. Porosity is observed to be inversely proportional to the two parameters studied. Higher moulding pressure and sintering temperature produced dense bodies and undesirable structure and pore system. It appears that strong and microporous membranes could be produced at a pressing force of 120-140 kN and sintering temperature of 1400-1500C However, the optimum conditions could be determined only through permeability studies. The pore size distribution were found narrow in the range 2-10 μm and porosity in the range of 13-48%, suitable for microfiltration . A typical example of pore size distribution for a sample prepared at 1600 C and 145 kN is shown in Figure 4 . Permeability studies could not be conducted due to problem of fixing the fragile ceramic membrane in the existing permeator cell.



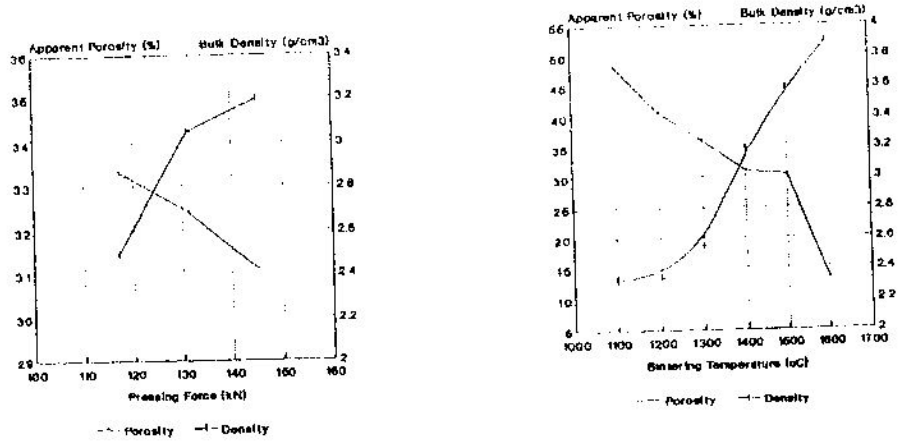
a) b)

Figure 1 : Microstructure Of Alumina Membrane at 115 kN Pressin Pressure and 1400 C Sintering Temperature . a) Top Surface Structure b) Cross sectional View.



a) b)

Figure 2 : Microstructure of Alumina Membrane at 145 kN Pressii Pressure and 1500 C Sintering Temperature. a)Top Surface Structu b) Cross Sectional View.



a) b)

Figure 3 : Effects of Operating Parameters On Membrane's Porosity A Density. a) Effect of Pressing Force. b) Effect of Sinter Temperature.

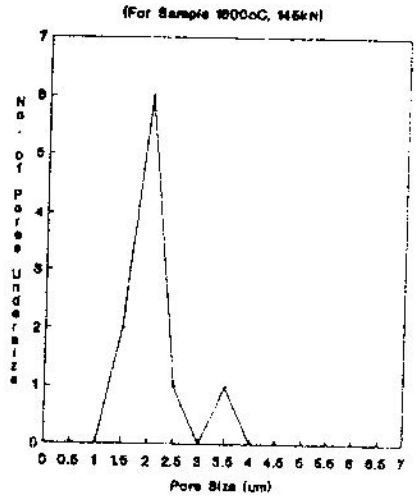


Figure 4 : Typical Example of A Pore Size Distribution For Alumina Membrane.

CONCLUSION

Ceramic alumina membrane sheets have been synthesized with a pore size range 2-10 μm and porosity of 13-48 % . These membranes can be used for microfiltration at elevated temperature and under extreme environmental conditions.They can be used as porous support for the production of composite-asymmetric UF/hyperfiltration membrane. Further work in the refinement of the pore size and permeation studies is envisaged.

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